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(54) Title: BEVERAGE PRODUCT WITH MODIFIED STARCH AND NITROGEN

(57) **Abstract:** A beverage product comprises a container holding a liquid beverage component and nitrogen gas; said liquid beverage comprising octenylsuccinic acid modified starch, and at least one surface active agent selected from the group consisting of acyl lactylate salts, proteins, protein hydrolysates, sucrose esters and mixtures thereof.

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BEVERAGE PRODUCT WITH MODIFIED STARCH AND NITROGEN

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The present invention relates to beverage products and in particular of foaming beverage products.

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There are many examples of foaming beverages which are produced by the use of inserts inside a pressurised can. In the United Kingdom many canned beers, stouts and lagers are sold in cans which contain a so-called "widget" which operates after the can

10 is opened to give a head on the drink which is said to be comparable to the head produced on draught drinks dispensed in bars. Examples of such widgets are described in GB-A-2183592, EP-A-360284, EP-A-577284, US-A-4996823, US-A-5009901, WO-A-9324384, WO-A-9504689. Examples of non-alcoholic pressurised
15 beverages which are pressurised with nitrous oxide and/or carbon dioxide are described in US-A-6403137 and GB-A-2299978.

Beverages that are packaged in a closed container in the presence of carbon dioxide or nitrous oxide and nitrogen are described in EP-A-745329 and EP-A-1034703. Foaming cappuccino
20 coffee products can be made by adding to the coffee drink a creamer comprising protein, lipid and carrier and optionally a modified starch emulsifier or a surfactant as is described in US-A-6168819. Effervescent beverages which are intended to be dispensed directly into the mouth of the consumer are described
25 in WO-A-02070371 and WO-A-02070372.

A first aspect of the present invention provides a beverage product comprising a container holding a liquid beverage component and nitrogen gas, said liquid beverage comprising

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octenylsuccinic acid modified starch, and at least one surface active agent selected from the group consisting of acyl lactylate salts, proteins, protein hydrolysates, sucrose esters and mixtures thereof.

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The container should be of sufficient strength that it can hold the pressure of the nitrogen gas inside it and should be impermeable to nitrogen gas. The container may be made of metal e.g., aluminium or steel, a plastic material for example
10 polyethylene terephthalate or glass. The pressure of the gas in the head space within the container should preferably be in the range 2 to 6 bar at 5°C. The term "nitrogen gas" as used herein is intended to include pure nitrogen gas or gas mixtures that are predominantly comprised of nitrogen. Preferably the
15 nitrogen gas has a purity of >97%.

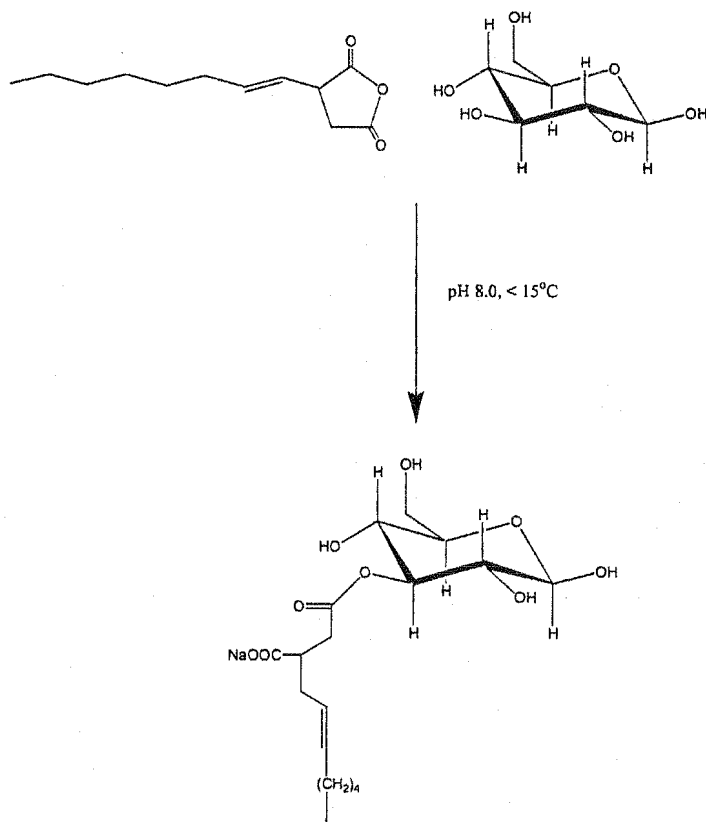
The liquid beverage component may be any consumable liquid. Examples of suitable liquids include optionally flavoured water, optionally flavoured milk, fruit flavoured liquids, tea or tea
20 flavoured liquids, coffee or coffee flavoured liquids, chocolate, chocolate flavoured liquids, fruit smoothies or alcoholic or alcohol-free drinks such as cream liqueurs or cocktails.

25 The nitrogen gas may be introduced into the container in the form of liquid nitrogen.

The octenylsuccinic acid modified starch may be prepared by forming a covalent complex of a hydrophilic waxy maize starch

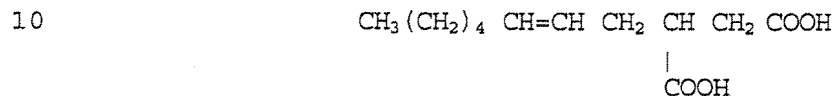
- 3 -

with an octenylsuccinic acid moiety preferably its anhydride. The production of the octenylsuccinic acid modified starch is shown in the reaction scheme below.



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Preferably the octenylsuccinic acid is a carboxy substituted undecenoic acid of formula



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ie 3-carboxy-undec-5-enoic acid

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The percentage molar substitution of octenylsuccinic acid groups may be in the range of 1.9 to 3%, preferably around 2.2%. The molecular weight of the octenylsuccinic acid modified starch is preferably in excess of 100,000 kDa.

5

The octenylsuccinic acid modified starch preferably comprises 0.25 to 3.0% more preferably 0.75 to 1.5% by weight of the liquid beverage component. Suitable octenylsuccinate acid modified starch include those available from National Starch
10 under the trade names Purity 2000, Purity 1773, Purity 539 and N-Creamer 46. A particularly preferred octenylsuccinic acid modified starch is available commercially from National Starch under the trade name N-Creamer 46

15 The viscosity of the liquid beverage component is preferably in the order of 1.5 to 100 mPa.s⁻¹, more preferably 30 to 60 mPa.s⁻¹ under low shear conditions (0.15 s⁻¹) at 5°C.

The acyl moiety of the acyl lactylate salt preferably contains 8
20 to 16 preferably 10 to 14 more preferably around 12 carbon atoms. The acyl lactylate salt may be a sodium or calcium salt. Preferred acyl lactylate salts include calcium stearyl lactylate and sodium stearyl lactylate and mixtures thereof. The acyl lactylate salt preferably comprises 0.005 to 1 %, more
25 preferably 0.01 to 0.5% by weight of the liquid beverage.

Suitable proteins and protein hydrolysates are those containing or derived from milk for example caseinate salts such as sodium caseinate, whey protein isolates or milk protein hydrolysates.

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The protein and/or protein hydrolysate preferably comprises 0.01 to 0.5 %, more preferably 0.1 to 0.3% by weight of the liquid beverage.

5 Sucrose esters are esters prepared from sucrose and fatty acids derived from edible fats and oils. Preferred sucrose esters are predominantly monoesters. The fatty acid moiety preferably contains 8 to 16 carbon atoms. Suitable fatty acids include caprylic acid, lauric acid, myristic acid, palmitic acid,
10 stearic acid and mixtures thereof. Suitable sucrose esters are commercially available from Ryoto under the trade names P-1570 (70% monoester with fatty acids derived from vegetable oils containing 70% palmitic acid) and M-1695 (80% monoester with fatty acids derived from vegetable oils containing 95% myristic
15 acid). The sucrose ester preferably comprises 0.02 to 0.4%, more preferably 0.05 to 0.3% of the liquid beverage.

In preferred beverage products of the present invention the surface active agent comprises an acyl lactylate salt either
20 alone or in combination with a sucrose ester, a protein or a protein hydrolysate.

The surface tension of the liquid beverage component should be in the order of 65 to 20 N.m⁻², more preferably 40-20 N.m⁻².

25

The beverages of the present invention may contain additional constituents. Examples of suitable additional constituents include:-

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- (a) sweeteners for example natural sweeteners such as sugars (glucose, fructose, sucrose or corn syrup) or artificial sweeteners such as saccharin, aspartame or acesulfam.
- (b) Preservatives for example benzoate or sorbate salts
- 5 (c) Antioxidants for example ascorbic acid or salts thereof or tocopherols
- (d) Flavour enhancers for example maltol
- (e) Flavourings for example fruit flavours or vanilla
- (f) pH adjusting agents for example sodium bicarbonate
- 10 (g) viscosity adjusting agents for example propylene glycol alginate, carboxymethyl cellulose, high methoxy pectin, and/or gums such as guar gum

A second aspect of the present invention provides a method of
15 making a beverage product comprising a container holding a liquid beverage component and nitrogen gas, said liquid beverage comprising octenylsuccinic acid modified starch, and at least one surface active agent selected from the group consisting of acyl lactylate salts, proteins, protein hydrolysates and sucrose
20 esters and mixtures thereof, said method comprising the steps of:-

- incorporating the octenylsuccinic acid modified starch and the at least one surface active agent into the liquid beverage,
- 25 placing the liquid beverage into the container,
- adding sufficient liquid nitrogen to the container to provide a head space pressure of 2 to 6 bar at 5°C in the container after sealing, and
- sealing the container.

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The contents of the sealed container may be sterilised after sealing by the application of heat for example by pasteurisation or retorting. Alternatively the product may be subjected to
5 microfiltration or may be filled aseptically.

The present invention provides a beverage which is retained under pressure inside the container before the container is opened but when the nitrogen becomes supersaturated after the
10 container is opened, comes out of solution and forms a stable foam on top of the liquid beverage. Preferably the volume of foam does not exceed 20% of the volume of the liquid beverage. The amount of foam may be enhanced by the inclusion in the can of a widget though the use of a widget is not essential to
15 achieve the foaming provided by the present invention. In preferred beverage products of the present invention no widget is required. The presence of the foam on top of the dispensed liquid beverage provides a pleasant drinking experience (eg a pleasant taste and creamy mouthfeel) to the consumer as the
20 beverage is consumed. The product may be consumed straight from the container, poured into a drinking vessel for example glass or sprayed directly into the mouth of the consumer.

The invention will be illustrated by the following non-limiting
25 examples

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Example 1

A milked tea beverage was made as described below.

- (1) Black tea leaf tea (0.6kg) was extracted with water (18L)
5 at $90 \pm 1^\circ\text{C}$ for 3 minutes. The infusion was then passed through a 20 mesh screen, followed by a 150 mesh screen and cooled to $20-30^\circ\text{C}$. The infusion was then clarified using a centrifuge.
- (2) Sugar (5.5kg) was dissolved in hot water (6L), sterilised
10 by UV treatment and added to the tea extract.
- (3) UHT-treated skimmed milk (10.6kg) was added to the resulting mixture
- (4) Sodium ascorbate (0.05kg) was dissolved in water (2L) and the solution added to the mixture.
- 15 (5) Water was added to a volume of 100L
- (6) The mixture was homogenised at $60-70^\circ\text{C}$ @ 200 kgf.cm^{-2} and heated to 85°C
- (7) Skimmed milk powder (1.106kg) was added and mixed at 13,500rpm for 2 minutes.
- 20 (8) Sodium stearyl lactylate (0.5kg) was added and mixed at 13,500 rpm for 2 minutes
- (9) N-Creamer 46 modified starch (1kg - ex National Starch) was added and mixed at 13,500 rpm for 2 minutes at 65°C .
- (10) The mixture was cooled to 10°C and maltol (0.03kg) was
25 added
- (11) The mixture (<295ml) was filled into standard 330ml beverage cans and sufficient liquid nitrogen was injected into the cans to give a head space pressure of $3.5 \pm 0.2 \text{ bar}$ at 5°C . The cans were then rapidly sealed.

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(12) The sealed cans were then retorted at 140°C for 5 minutes

The resulting beverage contained the following constituents

Constituent	Amount
Water	to 100%
UHT milk	10.60 %
Granulated sugar	5.5%
Tea solids	0.2%
Skimmed milk powder	1.16%
Tea flavour mix 06	0.16%
Sodium ascorbate	0.05%
Maltol	0.03%
N-Creamer 46	1.0%
Sodium stearyl lactylate	0.5%

5

Example 2

A tea beverage was made as described below.

- 10 (1) Leaf tea (0.65 kg) was extracted with water (90L) at 90 ± 1°C for 5 min. The infusion was then passed through 4 layers of muslin cloth and the temperature was held at 70°C.
- (2) Sodium bicarbonate (0.01 kg) was dissolved in the filtered infusion
- (3) Sugar (3.9 kg) was dissolved in the infusion at 70°C by
15 stirring gently for 1 minute.
- (4) Caramel (0.1kg) was added to the infusion at 70°C
- (5) Sodium stearyl lactylate (0.5kg) added and mixed at 13,500 rpm for 2 minutes

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- (6) N-Creamer 46 starch (1kg) added and mixed at 13,500 rpm for 2 minutes at 65°C
- (7) The resulting solution was cooled to 10°C
- (8) Maltol (0.03kg) was added
- 5 (9) Sodium ascorbate (0.05kg) was dissolved in water (2L) and added to the mixture
- (10) Tea aroma concentrate (2 kg) was added and the mixture was made up to 100l with water.
- 10 (11) The beverage mixture (<295ml) was filled into standard 330ml aluminium cans
- (12) Liquid nitrogen was injected in order to give a head space pressure of 3.5 ± 0.2 bar at 5°C and the cans were sealed rapidly.
- (13) The mixture was then retorted at 140°C for 5 minutes.

15

The resulting beverage contained the following constituents

Constituents	Amount
Water	to 100%
Tea solids	0.21%
Sugar	3.9%
Tea aroma concentrate	2.0%
Sodium ascorbate	0.05%
Sodium bicarbonate	0.01%
N-creamers 46	1.0%
Sodium stearoyl lactylate	0.5%
Maltol	0.03%
Caramel	0.1%

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Example 3

An Irish coffee-type beverage was made as described below.

- 5 (1) Water (81.2 kg) was heated to 75°C
- (2) Sugar (3.5kg) was added and completely dissolved at 70°C
- (3) A mixture of sodium stearyl lactylate (0.05kg), calcium
stearyl lactylate (0.05kg) and sucrose monoesters (0.2kg)
was added and mixed at 13,500 rpm at 70°C
- 10 (4) Skim milk powder (1.0kg) was added and mixed at 13,500 rpm
at 70°C
- (5) N-Creamer 46 (1.0 kg) was added and mixed at 13,500 rpm at
70°C
- (6) Instant coffee powder (0.8 kg) was added and dissolved at
15 60°C
- (7) The mixture was cooled to ambient temperature and whiskey
(12.2kg) was added
- (8) The beverage (<295ml) was placed in a standard aluminium
can (330ml) and sufficient liquid nitrogen was added to
20 give a head pressure of 3.5 ± 0.2 bar at 5°C and can was
sealed rapidly. Note. The product was filled and
nitrogenated under aseptic conditions.

The resulting beverage contained the following constituents

25

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Constituent	Amount
water	to 100%
sugar	3.50%
Sodium stearoyl lactylate	0.05%
Calcium stearoyl lactylate	0.05%
Sucrose monoesters	0.20%
Skimmed milk powder	1.0%
N-creamers 46	1.0%
coffee	0.80%
whiskey	12.20%

Example 4

5 A raspberry flavoured smoothie type beverage was made as described below.

- (1) Water (90 kg) is heated to 75°C
- (2) Sugar (4 kg) is added and completely dissolved at 70°C
- 10 (3) Sodium stearoyl lactylate (0.5 kg) is added and mixed at 13,500 rpm at 70°C
- (4) Skim milk powder (1 kg) is added and mixed at 13,500 rpm at 70°C
- (5) N-Creamer 46 (1 kg) is added and mixed at 13,500 rpm at
15 70°C
- (6) pH of solution is increased to pH 7.0 using 1.0M NaOH
- (7) Cooled to ambient temperature and raspberry juice (10 kg) is added. The pH of the solution is maintained at pH 6.5 with the addition of 1.0M NaOH

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(8) The beverage (<295ml) was placed in a standard aluminium can (330ml) and a commercially available widget was placed in the can.

(9) Sufficient liquid nitrogen was added to give a head pressure of 4 bar at 5°C and the can was sealed rapidly.

(10) The can was retorted at 121°C for 5min.

The resulting beverage contained the following constituents

10

Constituent	Amount
Water	to 100%
Raspberry juice	10%
Sugar	4%
N-creamer 46	1%
Sodium stearyl lactylate	0.5%
Skim milk powder	1%
Vanilla	0.05%

Example 5

A milked tea beverage was made as described below.

15

(1) Black tea leaf tea (0.6kg) was extracted with water (80L) at $90 \pm 1^\circ\text{C}$ for 3 minutes. The infusion was then passed through a 20 mesh screen, followed by a 150 mesh screen and cooled to 20-30°C. The infusion was then clarified using a centrifuge.

20

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- (2) Sugar (5.5kg) was dissolved in hot water (6L), sterilised by UV treatment and added to the tea extract.
- (3) UHT-treated skimmed milk (10.6kg) was added to the resulting mixture
- 5 (4) Sodium ascorbate (0.05kg) was dissolved in water (2L) and the solution added to the mixture.
- (5) Water was added to a volume of 90L
- (6) The mixture was homogenised at 60-70°C at 19.6kPa. [200 kgf.cm⁻²] and heated to 85°C
- 10 (7) Skimmed milk powder (1kg) was added and mixed at 13,500rpm for 2 minutes.
- (8) Sodium stearoyl lactylate (0.06kg) was added and mixed at 13,500 rpm for 2 minutes
- (9) N-Creamer 46 modified starch (1.25kg - ex National Starch)
- 15 was added and mixed at 13,500 rpm for 2 minutes at 65°C.
- (10) 0.2kg of milk protein hydrolysate (Hyfoama, ex. Quest) and dissolved thoroughly at 65°C
- (11) The resulting solution was cooled to 10°C and maltol (0.03kg) was added.
- 20 (12) The solution was made to 100L with water.
- (13) The mixture (<295ml) was filled into standard 330ml beverage cans and sufficient liquid nitrogen was injected into the cans to give a head space pressure of 3.5 ± 0.2 bar at 5°C. The cans were then rapidly sealed.
- 25 (14) The sealed cans were then retorted at 140°C for 5 minutes

The resulting beverage contained the following constituents

- 15 -

Constituent	% solids
Water	to 100%
UHT milk	10.60 %
Granulated sugar	5.5%
Sucrose esters (P1570)	0.1%
Hydrolysed milk protein (Hyfoama DS, Quest)	0.2%
Tea solids	0.2%
Skimmed milk powder	1%
Tea flavour mix 06	0.16%
Sodium ascorbate	0.05%
Maltol	0.03%
N-Creamer 46	1.25%
Sodium stearoyl lactylate	0.06%

5 Comparative Examples A and B

In a similar way to that described above in Example 3, samples of beverages which had the same constituents as Example 3 were prepared except that Comparative Example A did not contain any surface active agents and comparative Example B did not contain any octenylsuccinic acid modified starch. The products were stored at 5°C for 3 hours and were then opened and poured into a graduated glass vessel. The amount of foam generated as the beverage was poured was determined from the graduations on the glass vessel. The amount of foam expressed as a percentage of

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the volume of foam present immediately after pouring was determined periodically for the beverage of Example 3 and for both of the Comparative Examples A and B. The results are shown in the Table below

5

	Example 3	Example A	Example B
Foam volume	6.34%	6.66%	7.93%
Time (minutes)	Foam volume as % of volume at t_0		
2.5	100	100	100
5	100	75	100
10	100	50	60
15	100	50	44
20	100	50	20
30	95	40	20
40	90	35	12
60	90	25	8

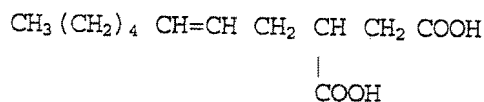
From the Table it can be seen that the foam generated from Example 3 lasts considerably longer than the foam generated from either of the Comparative Examples.

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CLAIMS

- 1) A beverage product comprising a container holding a liquid beverage component and nitrogen gas, said liquid beverage comprising octenylsuccinic acid modified starch, and at least one surface active agent selected from the group consisting of acyl lactylate salts, proteins, protein hydrolysates, sucrose esters and mixtures thereof.
- 2) A beverage product as claimed in claim 1 wherein the pressure of nitrogen in the head space of the container is in the range 2 to 6 bar at 5°C.
- 3) A beverage product as claimed in either of the preceding claims wherein the octenylsuccinic acid modified starch is prepared by forming a covalent complex of a hydrophilic waxy maize starch with an octenylsuccinic acid moiety
- 4) A beverage product as claimed in claim 3 wherein the octenylsuccinic acid is a carboxy substituted undecenoic acid of formula



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- 5) A beverage product as claimed in any one of the preceding claims wherein the percentage molar substitution of octenylsuccinic acid groups in the range of 1.9 to 3%.
- 5 6) A beverage product as claimed in any one of the preceding claims wherein molecular weight of the octenylsuccinic acid modified starch is in excess of 100,000 kDa.
- 10 7) A beverage product as claimed in any one of the preceding claims wherein the octenylsuccinic acid modified starch comprises 0.25 to 3.0% by weight of the liquid beverage component.
- 15 8) A beverage product as claimed in any one of the preceding claims wherein the acyl moiety of the acyl lactylate contains 8 to 16 carbon atoms.
- 20 9) A beverage product as claimed in any one of the preceding claims wherein the acyl lactylate salt is a sodium or calcium salt
- 25 10) A beverage product as claimed in any one of the preceding claims wherein the acyl lactylate salt is calcium stearoyl lactylate, sodium stearoyl lactylate or mixtures thereof.
- 11) A beverage product as claimed in any one of the preceding claims wherein the acyl lactylate salt comprises 0.005 to 1% by weight of the liquid beverage.

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- 12) A beverage product as claimed in any one of the preceding claims wherein the proteins and protein hydrolysates are those contained in or derived from milk.
- 5
- 13) A beverage product as claimed in any one of the preceding claims wherein the proteins and protein hydrolysates are selected from sodium caseinate, whey protein isolates or milk protein hydrolysates
- 10
- 14) A beverage product as claimed in any one of the preceding claims wherein the sucrose ester is predominantly a monoester.
- 15
- 15) A beverage product as claimed in any one of the preceding claims wherein the sucrose ester is prepared from sucrose and fatty acids derived from edible fats and oils, said fatty acids containing 8 to 16 carbon atoms
- 20
- 16) A beverage product as claimed in claim 20 wherein the fatty acid is caprylic acid, lauric acid, myristic acid, palmitic acid, stearic acid or mixtures thereof
- 25
- 17) A beverage product as claimed in any one of the preceding claims wherein the sucrose ester comprises 0.02 to 0.4% by weight of the liquid beverage.

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18) A beverage product as claimed in any preceding claim wherein the container also includes a widget.

19) A method of making a beverage product comprising a container
5 holding a liquid beverage component and nitrogen gas, said liquid beverage comprising octenylsuccinic acid modified starch, and at least one surface active agent selected from the group consisting of acyl lactylate salts, proteins, proteinhydrolysates and sucrose esters and mixtures thereof,
10 said method comprising the steps of:-
incorporating the octenylsuccinic acid modified starch and the at least one surface active agent into the liquid beverage,
placing the liquid beverage into the container,
15 adding sufficient liquid nitrogen to the container to provide a head space pressure of 2 to 6 bar in the container after sealing, and
sealing the container.

20

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP 03/12605

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A23L2/54 A23C9/152 A23F3/00 A23F5/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23L A23C A23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, FSTA, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 6 180 159 B1 (BUTTERBAUGH JEFFREY LEE ET AL) 30 January 2001 (2001-01-30) column 10, line 65 - line 67; claim 7 ----	1-25
A	QU Z H ET AL: "STARCH-BASED INGREDIENTS FOR FLAVOR ENCAPSULATION" CEREAL FOODS WORLD, AMERICAN ASSOCIATION OF CEREAL CHEMISTS, ST. PAUL, MN, US, vol. 44, no. 7, July 1999 (1999-07), pages 460-465, XP009005508 ISSN: 0146-6283 page 462, column 3 -page 464, column 1 ----- -/--	1-25

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

Z document member of the same patent family

Date of the actual completion of the international search

1 March 2004

Date of mailing of the international search report

19/03/2004

Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 03/12605

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>WEBER W: "GANZ IM SINNE DER VERBRAUCHER FORTSCHRITTE AUF DEM GEBIET DER SPEZIALSTAERKEN" LEBENSMITTELTECHNIK, HAMBURG, DE, vol. 29, no. 4, 1997, page 36 XP000971417 ISSN: 0047-4290 claims 2,3</p> <p style="text-align: center;">-----</p>	1-25

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information on patent family members

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